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<b>(21) International Application Number:</b> PCT/US92/07529 <b>(22) International Filing Date:</b> 1 September 1992 (01.09.92) <b>(71)(72) Applicant and Inventor:</b> SONG, Joo, H. [KR/US]; 1907 Butternut, Northbrook, IL 60092 (US). <b>(74) Agent:</b> SHURTZ, Steven, P.; William Brinks Olds Hofer Gilson & Lione, NBC Tower, Suite 3600, 455 North Cityfront Plaza Drive, Chicago, IL 60611 (US).		<b>(81) Designated State :</b> US.  <b>Published</b> <i>With international search report.</i> <i>Upon request of the applicant under Article 64(3)(c)(i).</i>
<b>(54) Title:</b> FLAVOR RELEASING STRUCTURES MODIFIED WITH PLASTICIZER FOR CHEWING GUM  <b>(57) Abstract</b>  An improved method of processing flavor releasing structures for use in chewing gum involves extruding a blend of thermoplastic material, non-thermoplastic material and a plasticizer. The flavor releasing structure may be coated with a flavor barrier coating.		

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FLAVOR RELEASING STRUCTURES  
MODIFIED WITH PLASTICIZER FOR CHEWING GUM

BACKGROUND OF THE INVENTION

The present invention relates to improved processing of chewing gum ingredients. More particularly, the invention relates to improving flavor-containing chewing gum ingredients by the use of specific plasticizers to give higher throughput of flavor releasing structures, better grinding and easier flavor loading. The improved chewing gum ingredient may also be used in a variety of chewing gum products such as sugar, non-sugar, and confectionery coated chewing gum products.

The present invention is an advance in the art of structures for use in chewing gums that gradually release a flavoring agent while the gum is chewed. These structures can provide improved flavor releasing characteristics during the latter portions of chewing and can provide greater flavor recovery than conventional means of delivering flavoring agents. Normally, gum containing a flavoring agent will exhibit a very strong, rapid flavor release in the beginning periods of chewing. After this initial flavor release, the flavor release dramatically decreases. The structures embodying the present invention can provide a large flavor release during the later periods of chewing, when the conventional gum's flavor release has diminished. Additionally, by varying the composition of the flavor releasing structure, different flavor release characteristics can be obtained.

U.S. Patent No. 5,128,155 discloses flavor releasing structures for chewing gum involving the use

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of a thermoplastic and a non-thermoplastic material. Several patents disclose ways of extending flavor release through the encapsulation of sweeteners using ethyl cellulose and a hydrophobic plasticizer. These patents are Australian Patent No. 87-081410; Japanese Patent No. 88-169938, and U.S. Patent No. 4,911,934.

U.S. Patent No. 4,502,888 discloses aqueous dispersions of cellulose derivatives using fatty acid salts as plasticizers.

U.S. Patent No. 4,568,560 discloses flavors and fragrances with controlled release that are encapsulated with ethylcellulose and plasticizer.

In U.S. Patent No. 4,931,286, a high gloss pharmaceutical tablet is prepared by spray coating the tablet with an aqueous solution of carboxymethyl - cellulose and a plasticizer.

#### SUMMARY OF THE INVENTION

The present invention is a method of improving the processing of a flavor releasing structure, subsequently incorporated into chewing gum, through the addition of a plasticizer. The flavor releasing structure comprises a flavor reservoir material, a binding material and a plasticizer. The flavor reservoir material can be a non-thermoplastic material. The binding material can be a thermoplastic material. The flavor reservoir material is dispersed in the binding material and is bound by it. These materials form the core of the flavor releasing structure. At least one of the materials releasably retains a flavoring agent.

#### DETAILED DESCRIPTION OF PREFERRED EMBODIMENT OF THE INVENTION

An embodiment of a structure made in accordance with the present invention is a chewing gum having flavor releasing structures made by extruding

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mixtures of thermoplastic, non-thermoplastic and plasticizer materials.

One of the support materials can be a thermoplastic. This material will deform or melt, becoming flowable at the temperatures and pressures used in the extruder. Another material used is a flavor reservoir material. This material is a non-thermoplastic, to the extent that it will not deform or melt at the temperature and pressure used in the extruder. A third material used is a plasticizer, which aids in the processing of the other two materials. The flavor releasing structure has at least these three materials that form a core of the structure.

In a process used to make the cores, the thermoplastic and non-thermoplastic materials and plasticizer are mixed together and added to a hopper which feeds the extruder. The increased temperature and pressure within the extruder cause the thermoplastic material to become flowable. In the past, the core materials would become tacky and cake inside of the extruder. The addition of the plasticizer prevents this by acting as an anticaking agent. Once the mixture has become flowable it is then forced through a die to form an extrudate. Because the mixture does not cake inside the extruder, there is a higher throughput of extrudate. The extrudate should be cooled immediately after extrusion. This can be accomplished by collecting the extrudate on a conveyor belt and cooling it by blowing air onto the extrudate. The extrudate is then ground and sieved if needed, to provide cores of a desired size. Extrudate with plasticizer is more brittle than extrudate without the plasticizer. This makes the grinding of the extrudate easier.

Various proportions of thermoplastic and non-thermoplastic materials can be used. Normally, when

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using a cellulosic material and silica, a maximum of 40% silica (based on the total weight of the mixture) can be used. A loading of as high as 60% silica is possible when using teflon as the thermoplastic material.

In addition to extrusion, other processes can be used to form the cores. For example, the materials can be combined in a heated pressure vessel, heated to a temperature at which the thermoplastic becomes flowable and mixed. Upon cooling, the resulting material can be ground and sieved if needed to provide cores of a desired size.

Grinding can be accomplished by means known in the art. For example, a Mikropul or Fitzmill grinder can be used. On large scale operations, where heat buildup may be a concern, cryogenic grinding can be used. Exposure of the extrudate to high humidity environment before or after grinding may reduce the core's ability to hold flavoring agent and may increase handling difficulties. By adding a plasticizer such as glycerol monostearate to the hydroxypropyl cellulose and silica, the grinding process of the extrudate is easier. The extrudate is more brittle than it would be without the glycerol monostearate, which makes it easier to grind.

The cores have at least two matrices. One matrix is comprised of the non-thermoplastic material and the other matrix is comprised of the thermoplastic material. The non-thermoplastic matrix is dispersed throughout the thermoplastic matrix. The thermoplastic material functions as a binding material for the non-thermoplastic material in the core. The thermoplastic material forms a matrix larger than the non-thermoplastic material and supports and incorporates the non-thermoplastic material. Thus, the non-thermoplastic material may be enclosed in the thermoplastic material to varying degrees. Some of the

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non-thermoplastic material may be completely enclosed in the thermoplastic material, some only partially enclosed in the thermoplastic material, and some may be substantially unenclosed in the thermoplastic material.

In addition to the two materials having different thermoplastic properties, they also can differ in their water solubility (how quickly they will dissolve in water). This difference may effect the release rates of the flavoring agent. The non-thermoplastic material should be essentially water insoluble. For example, silica can be used as the non-thermoplastic support material. On the other hand, the thermoplastic materials should be water soluble or, water swellable, but should have a relatively slow rate of dissolution. The water solubility of these materials will have an effect on the release rate of the flavoring agent. Generally, all other factors remaining the same, the higher the water solubility of the thermoplastic materials, the faster the release rates of the flavoring agent.

Cellulosic materials are preferred as the thermoplastic material. For example, cellulose-hydroxypropyl ether, which is called hydroxypropylcellulose, is particularly preferred. Hydroxypropylcellulose is sold by Aqualon Co., a subsidiary of Hercules Inc., under the trademark Klucel. Hydroxypropylcellulose is available in different molecular weights. For example, Klucel HF has a molecular weight of 1,500,000 and Klucel EF has a molecular weight of 80,000. Varying the molecular weight may affect the release characteristics of the core. Another example of cellulosic thermoplastic support material is hydroxypropyl hydroxyethylcellulose. This product is sold by Aqualon Co. under the trademark Natrovis. In addition to cellulosic materials, carbohydrates may also be used as the thermoplastic material. One aspect to consider in

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choosing a thermoplastic material is the degree to which the flavoring agent may plasticize the thermoplastic.

Thermosetting materials may also be used as one of the support materials in combination with, or in place of, the thermoplastic material.

The non-thermoplastic material must be capable of absorbing, adsorbing, retaining or serving as a reservoir for the flavoring agent. Nevertheless, the ability to retain the flavoring agent should not be so great that this material will not release the flavoring agent when used in a chewing gum. This can be accomplished by several types of materials. For example, the non-thermoplastic material can consist of small particles with micro-porous structures, it can consist of the compositions disclosed in U.S. Patent 4,497,832, it can consist of small particles with very high surface areas or it can contain a combination of any of these types of materials or properties. During processing, the non-thermoplastic material should not lose its ability to releasably retain the flavoring agent.

Examples of materials that can be used as flavor reservoir materials are silicas such as synthetic amorphous silica dioxide hydrate, which is supplied by Degussa Corp. under the tradenames Sipernat 22, 225, 50 and 503, and is also supplied by Cabot Corp. under the trademark Cab-O-Sil (EMS); microsponges, such as those supplied by Advanced Polymer Systems, Co.; zeolites; carbon black; and rubber particles either cross linked or not. Other silicas that can be used are those supplied by W.R. Grace Co. under the tradename Syloid silica. Preferably, Syloid silica is used due to the tendency of Sipernat silicas to cause oxidation of mint flavor during the storage of mint flavored gum. Porous



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materials such as maltodextrin and porous natural fiber can be used instead of silica.

Either hydrophilic or hydrophobic non-thermoplastic materials can be used. For example, flavor releasing structures were made with both hydrophilic silica and hydrophobic silica. The water affinity of the non-thermoplastic, however, affects the release rate of the flavor. All other factors being equal, the greater the water affinity, the greater the release rate.

The flavor releasing structure will also include a plasticizer. Preferably glycerol monostearate will be added to the thermoplastic and non-thermoplastic core ingredients. Besides glycerol monostearate, other plasticizers such as lecithin, mono and diglycerides, glyceryl lactopalmitate, sorbitan monostearate, triglyceride monostearate, octaglycerol monooleate, polysorbates 65, 60, and 80, acetylated monoglycerides, distilled monoglycerides, distilled propylene glycolmonoester and succinylated monoglycerides may be used.

By adding a plasticizer to the flavor core ingredients, the flavor loading process is much easier because the extrudate is less sticky.

The flavoring agent can be added to the cores before, during or after this formation. When the flavoring agent is added before extrusion, however, care must be taken to keep the temperatures low enough in the extruder to prevent the degradation of the flavoring agent. The success of these various orders of addition will depend on several factors, including the results sought to be obtained, the process conditions under which the core is formed, the materials used, and the flavoring agents used.

The flavoring agent can be added after formation by soaking the cores in a container of flavoring agents. Another method of adding flavoring

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agent is by misting the flavoring agent over the cores. Although these methods will work, it is preferred to obtain as homogeneous a distribution of the flavoring agent in the core as is possible. One such way to obtain a homogenous distribution is by using a Vee mixer. The Vee mixer has nozzles that mist the flavoring agent over the cores as they are tumbled. Vee mixers can be obtained from Patterson Industries (Canada) Limited. A Cone Shell Blender is another type of mixer that will achieve the desired uniform distribution of flavoring agent in the core. Although in most cases the flavoring agent will be absorbed into the cores very rapidly, it is desirable to allow the cores to mix for a sufficient time to reach equilibrium.

The flavoring agent is releasably retained by the cores to the extent that it is released when the gum containing the flavor releasing structures is chewed. Both the thermoplastic matrix and the non-thermoplastic matrix can releasably retain the flavoring agent. Additionally, different and multiple flavoring agents may be used in a single core. The cores can contain very large amounts of flavoring agent. For example, cores have been made that contained as much as 40 to 60% flavoring agent, based on the total weight of the core. Higher loadings are obtained when the flavoring agent is added to the core after it is formed then when it is added during formation. A level of 33% loading, based on the total weight of the core, is presently preferred. The higher the loading the faster the flavoring agent will be released from the core, all other factors being equal.

Flavoring agents may comprise essential oils, synthetic flavors, or mixtures thereof, including but not limited to oils derived from plants and fruits such as citrus oils, fruit essences, peppermint oil, spearmint oil, clove oil, oil of wintergreen, anise,

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and the like. Those skilled in the art will recognize that natural and artificial flavoring agents may be combined in any sensorially acceptable blend. All such flavors and flavor blends are contemplated by the present invention, including, by way of example, peppermint, spearmint, wintergreen, orange, blueberry, grape, strawberry, raspberry, lime, lemon, cherry, citrus, apple, pear, peach, plum, cola, licorice, cinnamon, menthol, clove, nut, vanilla, anise, hay, and eucalyptus. Flavoring agents are commercially available, for example, from Dragoco Inc., Firmenich Incorporated, Food Materials Corp., Fries & Fries, Fritzsche-Dodge & Olicott, Giandan Corp., Haarman & Reiner Corp., International Flavors & Fragrance, and Kalsec.

Once the flavoring agent is retained in the core, the core can be coated with a flavor barrier coating. The coating increases the shelf life of the flavor releasing structure when it is incorporated into a chewing gum. The coating also aids in handling and processing the cores by preventing the flavor from evaporating. The coating can prevent the flavor from migrating into the gum and can protect chemically sensitive flavoring agents from reacting with the ingredients of the gum. The coating can also affect the release rates of the flavoring agent.

Generally, any materials or synthetic material that exhibits a flavor barrier characteristic can be used for the coating. Coating materials that have a high affinity for the flavoring agent are less desirable because they may draw the flavoring agent out of the core. Inert materials and hydrophilic materials are preferred for the coating. For example, the coating can be waxes such as the following that are sold by Petrolite Corp.: Ultraflex, Victory, Be Square 175, Be Square 185, Be Square 195, Starwax 100, Petrolite L-700, Petrolite L-1030 and Mekon White;

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polymer and wax combination such as the following that are sold by Petrolite Corp.: Petrolite C-4040, CP-7, CP-11, CP-12 and Vybar 825, 260, 253, 103, 373; hydrophilic materials such as sugar, gelatins, gum arabic, algamato, chitosan, corn syrup, starch, cellulose (methyl-cellulose), carrageenan, polyvinyl pyrrolidone, polyvinyl alcohol, and ethylene vinyl alcohol copolymers (such as EVAL which is a tradename of Evalca Co.); polyvinyl acetates; and polyethylene. The cores can be coated once or multiple times with the same coating material or successively with different coating materials.

When choosing waxes as coatings, the higher melting point waxes such as Starwax 100 exhibit superior flavor barrier characteristics as compared to lower melting point waxes. Waxes with melting points in the range of about 135-200°F can be useful as a coating.

The coating can be added by a rotating disk coater, by a fluidized bed coater, spray chilling or by a tumbler. If the cores stick together prior to coating, silica can be added to break them apart. Once the cores have been coated, it may be necessary to further sieve the material to remove any agglomerated coating material or to further size the cores for use in a gum.

Once the flavor releasing structure has been formed it is then incorporated into a chewing gum. Addition to the gum mixer during the last stage of the mixing cycle is preferred to minimize any damage to the flavoring releasing structures. If a wax coating is used, the wax may change the texture of the gum, requiring modification of the gum base or formula.

Chewing gum consists of a gum base to which a water soluble bulk portion may normally be added. Chewing gum bases generally comprise a combination of

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elastomers and resins, together with plasticizers and inorganic fillers.

The gum base may contain natural gums and/or synthetic elastomers and resins. Natural gums include both elastomers and resins. Suitable natural gums include, but are not limited to, chicle, jelutong, sorva, nispero tunic, niger gutta, massaranduba balata, and chiquihul.

When no natural gums are used, the gum base is referred to as "synthetic" and the natural gums are replaced with synthetic elastomers and resins. Synthetic elastomers may include polyisoprene, polyisobutylene, isobutylene-isoprene copolymer (a copolymer from Exxon Corp. under the designation "butyl rubber"), styrene butadiene rubber, and the like.

The amount of elastomer used in the gum base can typically be varied between about 10 and about 20 percent depending on the specific elastomer selected and on the physical properties desired in the final gum base. For example, the viscosity, softening point, and elasticity can be varied.

Resins used in gum base may include polyvinyl acetate, polyethylene, ester gums, (resin esters of glycerol), polyvinyl acetate-polyethylene copolymers, polyvinyl acetate-polyvinyl laurate copolymers, and polyterpenes. Additionally, a polyvinyl acetate obtained from Monsanto under the designation "Gelva" and a polyterpene obtained from Hercules under the designation "Piccolyte" may be used.

As with the elastomer, the amount of resin used in the gum base can be varied depending on the particular resin selected and on the physical properties desired in the final gum base.

Preferably, the gum base also includes plasticizers selected from the group consisting of fats, oils, waxes, and mixtures thereof. The fats and oils can include tallow, hydrogenated and partially

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hydrogenated vegetable oils, and cocoa butter. Commonly employed waxes include paraffin, microcrystalline and natural waxes such as beeswax and carnauba. Additionally, mixtures of the plasticizers may be used, such as a mixture of paraffin wax, partially hydrogenated vegetable oil, and glycerol monostearate.

Preferably, the gum base also includes a filler component. The filler is preferably selected from the group consisting of calcium carbonate, magnesium carbonate, talc, dicalcium phosphate and the like. The filler may constitute between about 5 to about 60 percent by weight of the gum base. Preferably, the filler comprises about 5 to about 50 percent by weight of the gum base.

Further, gum bases may also contain optional ingredients such as antioxidants, colors, and emulsifiers.

These ingredients of the gum base can be combined in a conversational manner. In particular, the elastomer, resins, plasticizers, and the filler are typically softened by heating and then mixed for a time sufficient to insure a homogenous mass. The mass can be formed into slabs, or pellets and allowed to cool before use in making chewing gum. Alternatively, the molten mass can be used directly in a chewing gum making process.

Typically, the gum base constitutes between about 5 to about 95 percent by weight of the gum. More preferably, the insoluble gum base comprises between 10 and 50 percent by weight of the gum and most preferably about 20 to about 35 percent by weight of the gum.

In general, a chewing gum composition typically comprises a water soluble bulk portion added to the water insoluble chewable gum base portion. The flavoring agents are typically water insoluble. The water soluble portion dissipates with a portion of the

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flavoring agent over a period of time during chewing, while the gum base portion is retained in the mouth throughout the chew.

The water soluble portion of the chewing gum may further comprise softeners, sweeteners, flavoring agents and combinations thereof. Softeners are added to the chewing in order to optimize the chewability and mouth feel of the chewing gum. Softeners, also known in the art as plasticizers or plasticizing agents, generally constitute between about 0.5 to about 15.0 percent by weight of the chewing gum. Softeners contemplated by the present invention include glycerin, lecithin, and combinations thereof. Further, aqueous sweetener solutions such as those containing sorbitol, hydrogenated starch hydrolysates, corn syrup and combinations thereof may be used as softeners and binding agents in the chewing gum.

Sugar sweeteners generally include saccharide containing components commonly known in the chewing gum art which comprise, but are not limited to, sucrose, dextrose, maltose, dextrin, dried invert sugar, fructose, levulose, galactose, corn syrup solids, and the like, alone or in any combination. Nonsugar sweeteners can include sorbitol, mannitol, and xylitol.

Optional ingredients such as colors, emulsifiers and pharmaceutical agents may be added to the chewing gum.

In general, chewing gum is manufactured by sequentially adding the various chewing gum ingredients to a commercially available mixer known in the art. After the ingredients have been thoroughly mixed, the gum mass is discharged from the mixer and shaped into the desired form such as by rolling into sheets and cutting into sticks, extruding into chunks or casting into pellets.

Generally, the ingredients are mixed by first melting the gum base and adding it to the running

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mixer. The base may also be melted in the mixer itself. Color or emulsifiers may also be added at this time. A softener such as glycerin may also be added at this time, along with syrup and a portion of bulking agent. Further portions of the bulking agent may then be added to the mixer. The flavoring agent is typically added with the final portion of the bulking agent.

The entire mixing procedure typically takes about fifteen minutes, but longer mixing times may sometimes be required. Those skilled in the art will recognize that many variations of the above-described procedures may be followed.

The addition of a plasticizer to the thermoplastic and non-thermoplastic core ingredients works very well to increase the production rate of the extrudate. Because of its anticaking properties, a plasticizer causes the thermoplastic ingredient to be more flowable in the extruder.

Comparative  
Example 1

A mixture of 60% by weight Klucel HF and 40% by weight Sipernat 22 were extruded through a die having four holes each with a 3mm diameter. The extruder had seven temperature zones including the die. The first through sixth zones were at about 24°C, 59°C, 137°C, 200°C, 207°C and 212°C respectively. The die was at about 178°C. With the feeder setting at 60, this mixture was extruded at about 96 lbs of extrudate per hour.

Example 2

A mixture of 50% by weight Klucel HF, 30% by weight Sipernat 22, and 20% by weight glycerol monostearate was extruded through a die having 4 holes each with a 3mm diameter. The extruder had seven temperature zones including the die. The first through



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sixth zones were at about 25°C, 56°C, 142°C, 191°C, 194°C and 195°C respectively. The die was at about 171°C. This mixture was extruded at about 103.2 lbs of extrudate per hour.

Comparative  
Example 3

A mixture of 60% by weight Klucel EF and 40% by weight Sipernat 22 was extruded through a die having 4 holes each with a 3mm diameter. The extruder had seven temperature zones including the die. The first through sixth zones were at about 28°C, 55°C, 138°C, 142°C, 143°C and 144°C respectively. The die was at about 124°C. The mixture was extruded at about 40 lbs of extrudate per hour.

Example 4

A mixture of 50% by weight Klucel EF, 30% by weight Sipernat 22, and 20% by weight of glycerol monostearate was extruded through a die having 4 holes each with a 3mm diameter. The extruder had seven temperature zones including the die. The first through sixth zones were about 28°C, 51°C, 137°C, 138°C, 140°C and 143°C respectively. The die temperature was about 124°C. This mixture was extruded at about 150 lbs of extrudate per hour.

The inventive examples 2 and 4 were more brittle and had a higher throughput than the comparative examples 1 and 3 due to the addition of the glycerol monostearate, which served as a plasticizer.

Once the extrudate has cooled, it can then be ground to form cores that would pass through a 40 mesh screen. The cores can then be loaded with flavor up to a level of about 60% based on the total weight of the core and flavoring agent. To assure an even distribution of the flavoring agent in the cores, the flavor can be added to cores by using a Vee mixer. The cores can then be coated with wax or other sufficient coating by a rotating disk coater in a ratio of about 1

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to 1 cores to wax. The cores can be sieved again until particles become small enough to be incorporated into chewing gum.

It should be appreciated that the compositions and methods of the present invention are capable of being incorporated in the form of a variety of embodiments, only a few of which have been illustrated and described above. The invention may be embodied in other forms without departing from its spirit or essential characteristics. The described embodiments are to be considered in all respects only as illustrative and not restrictive, and the scope of the invention is, therefore, indicated by the appended claims rather than by the foregoing description. All changes which come within the meaning and range of equivalency of the claims are to be embraced within their scope.

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CLAIMS:

1. A method of preparing a flavor releasing structure for the use in chewing gum comprising the steps of:

- a) preparing a blend comprising about 30 to about 95 weight percent thermoplastic material, about 0 to about 50% weight percent non-thermoplastic material, and about 5 to about 40 weight percent plasticizer;
- b) melting the thermoplastic material and mixing the thermoplastic material, non-thermoplastic material and plasticizer in an extruder to form a flavor releasing product;
- c) extruding the flavor releasing product;
- d) grinding the flavor releasing product; and
- e) flavor loading the flavor releasing product.

2. The method of Claim 1 wherein the thermoplastic material comprises a cellulosic material.

3. The method of Claim 1 wherein the thermoplastic material comprises a carbohydrate.

4. The method of Claim 1 wherein the non-thermoplastic material comprises a silica.

5. The method of Claim 1 wherein the plasticizer is selected from the group consisting of glycerol monostearate, lecithin, mono and diglycerides, glyceryl lactopalmitate, sorbitan monostearate, triglyceride monostearate, octaglycerol monooleate, polysorbates 65, 60, and 80, acetylated monoglycerides, distilled monoglycerides, distilled propylene glycolmonoester, succinylated monoglycerides, fats, oils, waxes, and combinations thereof.

6. The method of Claim 1 wherein the thermoplastic material comprises hydroxypropyl cellulose, the non-thermoplastic material comprises silica, and the plasticizer comprises glycerol monostearate.

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7. The method of Claim 1 further comprising the step of coating the flavor releasing structure with a flavor barrier coating.

## INTERNATIONAL SEARCH REPORT

PCT/US92/07529

**A. CLASSIFICATION OF SUBJECT MATTER**

IPC(5) :A23G 3/30

US CL :426/5

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 426/3,4 AND 6,426/5

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US,A, 4,981,698 (CHERUKURI ET AL), 01 JANUARY 1991.	1-7
E,A,&	US,A, 5,165,944 (SONG ET AL), 24 NOVEMBER 1992.	1-7
A	US,A, 5,116,627 (RUTHERFORD ET AL), 26 MAY 1992.	1-7
A,&	US,A, 5,128,155 (SONG ET AL.), 07 JULY 1992.	1-7
A	US,A, 4,568,560 (SCHOBEL), 04 FEBRUARY 1986.	1-7
A	US,A, 4,931,293 (CHERUKURI ET AL), 05 JUNE 1990.	1-7

☒ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:	*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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Date of the actual completion of the international search

03 DECEMBER 1992

Date of mailing of the international search report

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Name and mailing address of the ISA/  
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## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US92/07529

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US,A, 4,931,295 (COURTRIGHT ET AL.), 05 JUNE 1990.	1-7
A	US,A, 4,933,190 (CHERUKURI ET AL). 12 JUNE 1990.	1-7